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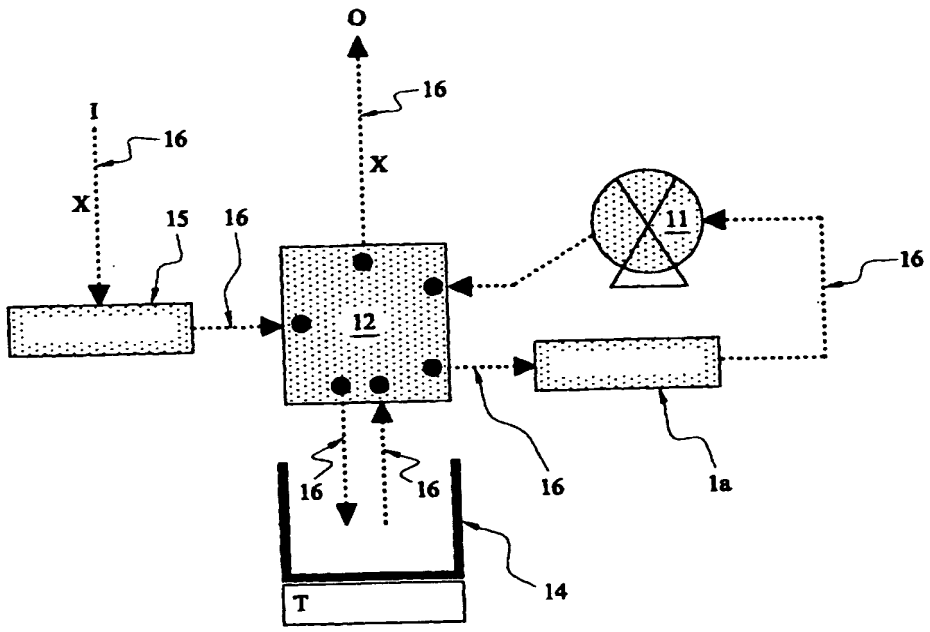

(57) Abstract: A chemical sensing system for gas or vapour analysis of a sample of interest, in particular to an array-based chemical sensing system for gas or vapour headspace analysis of food packaging materials.

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CHEMICAL SENSING SYSTEM

The present invention relates to a chemical sensing system for gas or vapour analysis of a sample of interest, in particular to an array-based chemical sensing system for gas or vapour headspace analysis of food contact packaging materials.

Current legislation requires that materials and articles that come into contact with food must not transfer their constituent chemicals to the food in quantities that would either endanger health or cause the food to become tainted or odorous. By way of example, chemicals which cause packaging to become tainted or odorous packaging may migrate into the food placed within the packaging. Food products such as chocolate and tea are particularly sensitive to taint and odour. Manufacturers of food contact packaging are therefore required to undertake "taint and odour" testing in order to observe stringent quality control procedures defined by current legislation and to meet the requirements of their customers.

Although the analytical chemist has available to him an extensive range of conventional instrumentation, quality control techniques are generally confined to the remote laboratory under the direction of trained personnel. Currently food contact packaging manufacturers rely in general on human sensor panels (HSPs) and/or gas chromatography/mass spectrometry (GC/MS) for quality control. HSPs have remained the industry standard for "taint and odour" testing for over two decades but suffer from two major disadvantages: (1) HSPs are subjective and cannot be calibrated against other panels (and are therefore prone to

error) and (2) HSPs are an off-line technique providing results 36 hours or more post production. Whilst being an accepted analytical technique, GC/MS remains a remote procedure and GC/MS devices are expensive to install and run and are operable only by trained operators.

There is an increasing demand to improve quality control procedures and to reduce the costs attributable to production mistakes. Bringing quality control techniques to the factory floor using technology for non-skilled operators is an extremely important goal to many manufacturing industries. For example, if an analytical technique is to provide added value to a carton manufacturer's quality control procedures, it should be able to provide reliable quality control information in a production environment.

The present invention seeks to fulfil the demand for improvements in quality control by providing an array based chemical sensing system. More particularly, the present invention provides a chemical sensing system comprising a modular sampling unit adapted such that the majority of the surface area of a sample (*eg* a paperboard carton or related raw material) is exposed to a continuous flow of a carrier gas (*eg* air) whilst minimising the sampling dead volume operatively connected to an array based chemical sensor assembly which exhibits improved sensitivity. Whilst there is little or no sample preparation before use, the chemical sensing system is able to provide rapid results at-line rather than post-production.

Thus viewed from one aspect the present invention provides a chemical sensing system for analysing the

headspace of a sample comprising a modular sampling unit operatively connected to a chemical sensor assembly, wherein:

(1) said modular sampling unit comprises:

a mount having an inlet channel and an outlet channel for a carrier gas;

a base member having a hollow interior bound by one or more internal side walls and a basal wall, wherein at least a part of the juncture of the basal wall and one or more internal walls is provided with a supporting collar for supporting the whole or part of the edge of the lower face of the sample;

a closure member having one or more exterior side walls and a basal wall defining a body portion, said body portion being complimentarily shaped with the hollow interior of the base member, wherein the outer edge of the basal wall is provided with an upstanding portion capable of engaging the edge of the upper face of the sample,

wherein in use the base member is inserted into the mount and the body portion of the closure member engages the hollow interior of the base member thereby defining a headspace below the lower face of the sample and a headspace above the upper face of the sample such that the inlet channel communicates with the headspace below the lower (or with the headspace above the upper) face of the sample and the outlet channel communicates with the headspace above the upper (or with the headspace below the lower) face of the sample thereby defining a continuous flow path between the inlet and outlet channel across the majority of the surface area of the sample; and

(2) said chemical sensor assembly comprises:

one or more chemical sensors, each chemical sensor having a chemical sensing component capable of exhibiting a

measurable and characteristic response to a chemical stimulus;

a solid body having an entry end for admitting a carrier gas, an exit end for exhausting a carrier gas and one or more compartments for housing each of the one or more chemical sensors therebetween, said one or more compartments being in consecutive fluid communication so as to define a continuous flow path between the entry end and the exit end of the solid body in which the chemical sensing component of the or each chemical sensor may be exposed to the carrier gas.

In use, a carrier gas is introduced into the inlet channel of the modular sampling unit where it sweeps the headspace of the sample. The carrier gas (containing one or more chemical stimuli from the headspace) is then drawn over each chemical sensor in the array and interacts to varying extents with each chemical sensor to yield a time dependent response profile which is characteristic of the chemical stimuli. Statistical methods for defining X% confidence intervals on multi-variate sample populations in conjunction with data reduction techniques may be used to draw conclusions as to the identity of the unknown chemical stimulus. The analytical results may be obtained as simple or as complex as desired (*ie* a simple pass/fail answer with or without a certainty value or conclusions may be drawn as to the possible identity of the chemical stimuli).

In a preferred embodiment, the hollow interior of the base member and the body portion of the closure member are substantially cylindrical. In this embodiment, the supporting collar and/or the upstanding portion are substantially annular. The annular supporting collar may

contain one or more cut-away portions. The annular upstanding portion may be a solid ring.

In a preferred embodiment, the inlet channel is connected to the headspace below the lower face of the sample by a basal conduit in the basal wall of the base member. The base member is inserted into the mount such that the inlet channel and basal conduit are in fluid communication.

In a preferred embodiment, the outlet channel is connected to the headspace above the upper face of the sample by a side conduit in the side wall of the base member. The base member is inserted into the mount such that the outlet channel and side conduit are in fluid communication. Particularly preferably the headspace above the upper face of the sample and side conduit are connected in fluid communication by a linear tube disposed radially in the upstanding portion of the closure member. The upstanding portion may contain more than one linear tube to accommodate samples of varying thickness by allowing the extent to which the body portion of the closure member operatively engages the hollow interior of the base member to be varied.

In a preferred embodiment, the headspace below the lower face of the sample is in fluid communication with the headspace above the upper face of the sample via one or more substantially U-shaped tubes. Particularly preferably a first arm of the (or each) U-shaped tube is disposed radially in the supporting collar of the base member and a second arm of the or each U-shaped tube is disposed radially in the upstanding portion of the closure member, the first and second arm being connected in fluid communication by a

connecting portion in the side wall of the base member. The supporting collar of the base member may contain more than one first arm and the side wall may contain more than one connecting portion so as to accommodate samples of varying thickness by allowing the extent to which the body portion of the closure member operatively engages the hollow interior of the base member to be varied.

In a preferred embodiment, the basal wall of the base member comprises a protrusion for supporting the lower face of the sample. Preferably the protrusion is capable of supporting the substantially central region of the lower face of the sample. Preferably the protrusion converges to a reduced contact end (*eg* to a point) so as to maximise exposure of the lower face of the sample to the carrier gas. Preferably the protrusion is substantially cone-shaped.

In a preferred embodiment, the basal wall of the closure member comprises a protrusion for assisting sample location and retention. Preferably the protrusion converges to a reduced contact end (*eg* to a point) so as to maximise exposure of the upper face of the sample to the carrier gas. Preferably the protrusion is substantially cone-shaped.

Preferably the body portion of the closure member is threadedly engaged within the hollow interior of the base member. Preferably the base member is a tight push fit into the mount.

Preferably the exterior face of the basal wall of the base member and the mount comprise a locating arrangement to enable correct positioning and insertion of the base member in the mount. For example, the locating arrangement may

comprise a locating pin on the mount capable of cooperating with a locating aperture on the exterior face of the basal wall of the base member.

Preferably the inlet and outlet channels are sealingly isolated (*eg* using one or more O-ring seals in the mount). Preferably the inlet channel is sealingly isolated from the environment (*eg* using one or more O-ring seals in the mount). Preferably the outlet channel is sealingly isolated from the environment (*eg* using one or more O-ring seals in the mount). Preferably the headspace of the sample and the environment are sealingly isolated (*eg* using one or more O-ring seals in the closure member).

The sampling chamber defined when the base member is inserted into the mount and the body portion of the closure member engages the hollow interior of the base member may be tailored to conform to the characteristics of the desired sample (*eg* solid), to the sample size and/or shape and to considerations such as destructive or non-destructive sampling. Preferably, the sampling unit is adapted to define a sampling chamber for use with solid samples.

Preferably each compartment of the solid body is arranged such that the chemical sensing component is exposed to the carrier gas (containing the chemical stimuli from the headsapce) in a wall jet fashion. The "wall-jet effect" is generally known from the art of liquid dynamics where studies have been made on the effects of liquid impingement on an extended solid surface.

In a preferred embodiment, the entry end for admitting a carrier gas and the exit end for exhausting a carrier gas

are connected by a substantially linear conduit. Preferably the substantially linear conduit defines a spine connecting each of the compartments consecutively. Particularly preferably each compartment is connected in parallel spaced apart relationship. Particularly preferably each compartment is substantially perpendicular to the spine. Particularly preferably each of the compartments is symmetrically arranged around the spine. In this manner, the carrier gas passes into each compartment where it impinges on the chemical sensing component in a wall-jet fashion (*ie* a divergent flow path is created in each compartment causing the carrier gas to traverse the surface area of the chemical sensing component) in a repeatable manner.

In a preferred embodiment, each compartment is adapted to house the chemical sensing component in substantially free space. In this embodiment, the chamber is sized and configured in accordance with the size and shape of the chemical sensing component and to minimise the dead volume.

Preferably the chemical sensor assembly comprises a plurality of chemical sensors (*ie* an array). Array based sensing systems have been the subject of much research over the last fifteen years (see for example Gardner *et al*, Sensors and Actuators B, 1994, 18 to 19, 211; and Grate *et al*, Anal. Chem., 1988, 60, 2801) and their properties are in general familiar to those skilled in the art.

Preferably the chemical sensing component is a planar chemical sensing component. The planar chemical sensing component may be any convenient shape and of any convenient type as desired for the stimulus of interest. For example, each chemical sensing component may be of a bulk or surface

acoustic wave type, a metal oxide type, a conducting polymer type or an optical type. Such chemical sensing components are widely reported and well-known (*eg* optical sensor components in WO-A-98/22807). Preferred are quartz crystal chemical sensors (*ie* a bulk acoustic wave type sensor).

Generally but not essentially, one or both faces of the planar chemical sensing component is coated with a material capable of exhibiting or inducing a measuring response to the stimulus of interest. Materials and methods for coating sensor components are familiar to those skilled in the art and are widely reported such as in for example King, Anal Chem, 1964, 36, 1735.

The materials of the chemical sensor assembly are adapted to resist absorption of the carrier gas and to minimise cross-contamination. For example, the solid body is conveniently constructed of an inert material (such as PTFE). The chemical sensor assembly may be directly mounted on the electronic circuitry board. This has the added advantage that leads between the sensing assembly and electronic circuitry are eliminated so that sensitivity (*ie* signal to noise ratio) is enhanced.

The chemical sensor assembly is itself novel and improves the sensitivity of gas and vapour analysis by more effectively exposing chemical sensors to a carrier gas.

Viewed from a further aspect the present invention provides a chemical sensor assembly as hereinbefore defined.

The modular sampling unit is itself novel and improves the sampling operation by exposing the majority of the

surface area of a sample to a substantially uninterrupted flow of carrier gas (*eg* air) in a low dead volume. The advantage of this is that the concentration of the stimulus of interest in the carrier gas is optimised thereby enhancing overall sensitivity.

Viewed from a yet further aspect the present invention provides a modular sampling unit as hereinbefore defined.

The chemical sensing system of the invention is suitable for use in any application where gas or vapour phase analysis of a sample is desirable. For example, the invention may be used to screen for volatile components in food contact packaging (*eg* printed paperboard cartons, paper or related materials) or in textiles. For this purpose, the chemical sensing system preferably comprises a template for tailoring the sample to the size and configuration of the sampling unit.

Viewed from a yet still further aspect the present invention provides a method for detecting the presence of one or more chemical stimuli in the headspace of food packaging material using a chemical sensing system as hereinbefore defined, said method comprising:

inserting the base member into the mount;

positioning a sample of the food packaging material in the hollow interior of the base member such that the sample is supported on the supporting collar;

engaging the body portion of the closure member with the hollow interior of the base member;

sweeping the headspace of the sample with a carrier gas;

passing the carrier gas containing one or more chemical stimuli from the headspace to the entry end of the chemical sensor assembly;

measuring the response of the chemical sensors to the carrier gas containing the one or more chemical stimuli from the headspace; and

relating the response to the presence of one or more chemical stimuli.

The chemical stimulus of interest may be (for example) diisopropylnaphthalenes (DIPNs) and the method may be used to rapidly detect DIPNs in paperboard at the levels of current concern to the industry.

The chemical stimulus of interest may be (for example) hexanal and the method may be used to rapidly detect hexanal contained in paperboard at levels which are indicative of potential problems for food packaging.

The chemical sensing system of the invention and its component parts may be controlled using suitable expert software. The expert software may be adapted to control the process and analyse data instantaneously to permit use by non-technical operators. Equally, the chemical sensing system may support auto-checking procedures to track chemical sensors and components reliably and to implement calibration of the chemical sensor component.

The present invention will now be described in a non-limitative sense with reference to the accompanying Figures in which:

Figure 1 illustrates a cross-sectional view of an array based chemical sensor assembly of the invention;

Figure 2 illustrates in detail an individual sensor of the array based chemical sensor assembly shown in Figure 1;

Figure 3 illustrates a side view of an array based chemical sensor assembly of the invention;

Figure 4 illustrates schematically the control system of an array based chemical sensor assembly of the invention;

Figure 5 illustrates schematically the control system of an embodiment of the modular sampling unit of the invention;

Figure 6 illustrates schematically the gas flow through an embodiment of the chemical sensing system of the invention;

Figure 7 illustrates (a) the mount, (b) the base member and (c) the closure member of a disassembled embodiment of the modular sampling unit of the invention;

Figure 7d illustrates an assembled embodiment of the modular sampling unit of the invention; and

Figures 8 and 9 illustrate the results of test Examples of an embodiment of the chemical sensing system of the invention.

Figure 1 illustrates in cross-sectional view an embodiment of a chemical sensor assembly of the invention designated generally by reference numeral 1. The chemical sensor assembly comprises an array of eight individual quartz crystal gas sensors 2 for bulk acoustic wave sensing, each sensor 2 having a planar chemical sensing component 2a. Each sensor 2 is housed in one of a series of compartments 50 of a solid sensor block 1a made of an inert material such as PTFE. The sensor block 1a has a linear conduit 51 between entry and exit ends which interconnects the series of compartments 50 in parallel spaced apart relationship thereby defining a carrier gas flow path X in which each

planar chemical sensing component 2a is disposed so as to permit gas flow over its surface in a unique manner (see Figure 2).

Figure 2 illustrates in detail the flow path X of a carrier gas over the surface of the planar chemical sensing component 2a of the quartz crystal sensor 2. "Wall jet" impingement onto the chemical sensing component 2a leads to a divergent flow path around and over its entire surface thereby contributing to an overall improvement in sensitivity.

Figure 3 illustrates in side view the sensor block 1a connected directly to the electronic circuitry 3 (the combined unit being referred to as a sensor board 4).

The sensor board 4 is controlled by a microcontroller 6 (see Figure 4) which takes instructions directly from a personal computer C down an RS232 line 9. The microcontroller 6 controls each sensor transducer 5, drive and output, and the temperature of the sensor block using a feedback mechanism 7, 8. The temperature control 7 permits heating and cooling of the sensor block.

As illustrated in Figure 5, a control board 10 for a sampling unit 14 is controlled by a microcontroller 13 from a personal computer C taking instructions down an RS232 line 9. Commands may be issued from the personal computer C to the microcontroller 13 during use or using whole sampling routines which are downloaded and subsequently executed. The control board 10 controls the switching of a pump 11 and a series of valves 12 and the temperature of the sampling chamber 14. The control board 10 may also support a series

of LEDs or an LCD panel to indicate the status of the instrument.

With reference to Figure 6, during use a carrier gas such as air (or a vapour) is drawn along flow path X from an inlet I to an exhaust O via the sampling unit 14 and the sensor block 1a. Flow is achieved by virtue of a pump 11 and a series of valves 12 together with connecting pipework 16. The valves 12 and pump 11 are controlled by a microcontroller 13 (see Figure 5) which allows the carrier gas or vapour to be directed around the system and allows filtered air to be drawn through filter 15 and over the chemical sensors after use for the purposes of cleaning. The wetted material of the pump 11, series of valves 12 and connecting pipework 16 are chemically inert. The sampling unit 14 is provided with temperature control T.

Figures 7a, b and c illustrate a cross-sectional view of the disassembled components of a modular sampling unit of the invention. Figure 7a shows a mount 17 into which the base member of the sampling unit is engaged by a push-fit (described hereinafter). Channels 18a partially define the flow path X of a carrier gas. Correct location of the mount 17 with the base member of the sampling unit is aided by a locating pin 20. The gas tight seal between the mount 17 and the base member and the separation of the gas inlet and gas outlets is achieved by three O-rings 19.

With reference to Figure 7b, a base member 21 comprises a locating pin hole 22 which engages the locating pin 20 of the mount 17 into which the sampling base 21 is inserted by a push-fit. A sample (for example paperboard) is cut to a predetermined size using a template and is placed into the

base member 21 where it is supported on internal walls 24a. Channels 18b align with channels 18a of the mount 17.

Illustrated in Figure 7c is the closure member 25. The closure member 25 is secured to the base member 21 by means of a screw thread 23b on the body portion 54 engaging an internal thread 23a on the base member. An O-ring 26 provides a gas tight seal between the base member 21 and the closure member 25. The basal wall of the closure member 25 comprises upstanding portions 24b which engage the uppermost surface of the sample.

Each of the base member 21 and closure member 25 are manufactured from inert materials and/or from materials which enable the chamber to be disposable or alternatively which allow cleaning and re-use.

Figure 7d shows a fully assembled modular sampling unit of the invention in which the base member 21 is inserted in the mount 17 and the closure member 25 is screwed into the base member 21. The sample is supported on internal walls 24a of the base member and the upper surface is sealingly engaged by upstanding portions 24b. A U-shaped tube 56 is formed during assembly to complete the continuous path X for flow of carrier gas across the majority of the exposed surface area of the sample. In this manner, a carrier gas may be used to sweep the headspace of the sample in an effectively low dead volume.

Example 1 - Demonstration of "at-line" Analysis

Experimental

Various carton samples of a single production run were analysed using an embodiment of a chemical sensing system according to the invention. Samples were taken at different post-production times (including immediately post-production, at 14 hours post-production and at numerous intervals therein). 50 x 60 mm squares were cut from the board and no further sample preparation was undertaken.

Results

The combined chemical sensor responses to replicate carton samples of a duplicate production run were used to define a qualitative calibration. The "calibration" cartons had been previously certified to be "within specification" using an alterative analytical technique. The various production carton samples were compared to this calibration.

Table 1

Sample	Probability (%)	Status	Comments
0 hrs / 1	0.12	Reject	Immediate
0 hrs / 3	3.4	Reject	5 mins post-prod
0 hrs / 5	9.8	Accept	10 mins post-prod
0 hrs / 6	29.9	Accept	12 mins post-prod
4 hrs	21.6	Accept	Post-air
14 hrs	39.6	Accept	Current anal. time

Discussion

The carton samples that were taken straight from the press (0 hrs/1-6) demonstrate a gradual increase in certainty of belonging to the "standard" population. Whilst

sample 0 hrs/1 which was analysed immediately, is rejected from the "standard" population, sample 0 hrs/5 analysed 10 minutes post-production, is accepted as belonging to the "standard" population. This is almost certainly due to the rapid loss of residual solvents from the carton.

Conclusions

These results demonstrate that cartons could be analysed almost immediately post-production thus eliminating the current 14 hour "airing" period and 24 hour incubation period before carton samples can be checked for quality. In addition, production can be monitored during progress thus yielding added value to the manufacturer's quality procedures.

Example 2 - The Detection of hexanal in Paperboard

The presence of hexanal in raw paperboard is often perceived to be a marker of potential quality problems in the resulting packaging.

Experimental

Two samples of paperboard packaging were analysed using an embodiment of the chemical sensing system according to the invention. One sample was of "acceptable" packaging, the other had been "rejected" by a HSP. 50 x 60 mm squares were cut from the packaging and no further sample preparation was undertaken.

Results

The combined chemical sensor responses to replicate samples of the "acceptable" packaging were used to define a qualitative calibration. This calibration was used to compare the "rejected" packaging (Figure 8).

Discussion

Figure 8 demonstrates that the embodiment of the chemical sensing system according to the invention is clearly able to resolve low levels (< 0.8 ppm) from higher levels (1.6 ppm) of hexanal contained in paperboard. The levels of hexanal contained in the paperboard were independently determined using a traditional analytical technique. Investigations into other contaminants found in paperboard have demonstrated that the embodiment of the chemical sensing system according to the invention is able to provide reliable quantitative information over wide dynamic ranges.

Conclusions

The embodiment of the chemical sensing system according to the invention provides a rapid (< 2 minutes) technique to determine the hexanal content of paperboard which is of utility to both paperboard producers and to packaging manufacturers.

Example 3 - The Detection of a Contaminated Food Contact Carton

Experimental

A "tainted" food contact carton sample was analysed using an embodiment of the chemical sensing system according to the invention. Samples of various cartons that were "in specification" were also analysed using the embodiment of the chemical sensing system according to the invention to provide the instrument calibration. 50 x 60 mm squares were cut from the board and no further sample preparation was undertaken.

Results

The combined chemical sensor responses to the "in specification" carton samples were used to define a qualitative calibration. The "tainted" food contact carton was compared to this calibration and the results are given in Table 2.

Table 2

Sample Name	Probability (%)	Status	Comments
T	0	Reject	Tainted carton
U	0	Reject	paperboard layer
V	7.1	Accept	Print layer of T

Discussion

The results of the sample comparisons to the "standards" calibration are presented as percentage probabilities of belonging to the "standards" population. The customary 5% significance level was applied as the pass/ fail boundary. The results identified the "tainted" carton

(T) as contaminated. This carton (T) had previously been analysed using a HSP and had "passed". It was later identified by a second HSP as being "tainted". This conflict of results draws attention to the subjective nature of the HSP technique. The "tainted" carton (T) was divided into its two component layers and re-analysed. The results suggested that the source of taint arose from the paperboard (sample U). The chemicals which gave rise to the taint may have originated from the board or may have been the result of a chemical reaction in the board during production (probably initiated by the UV light). It has now been confirmed by GC-MS that the conclusions reached by the embodiment of the chemical sensing system according to the invention were in fact correct and that it was the paperboard that was the source of contamination.

Conclusions

This embodiment of the chemical sensing system according to the invention was able to successfully identify a carton as "tainted" which a human sensory panel was unable to detect.

Example 4 - The Detection of DIPN in Paperboards Intended for Food Contact

Diisopropylnaphthalenes (DIPNs) have been found in both paperboard and paperboard packaging intended for food contact. It is evident that DIPNs can migrate into the food which they package. The full toxicological effects of DIPNs are currently unknown and manufacturers of paperboard and paperboard packaging have been advised to keep the levels of DIPNs in packaging as low as is practical until such time

that more information is available on the toxicological effects of DIPNs.

Experimental

Six samples of paperboard were analysed using an embodiment of the chemical sensing system according to the invention. 50 x 60 mm squares were cut from the board and no further sample preparation was undertaken. The embodiment of the chemical sensing system according to the invention was used in a method consisting of a 25 second sampling time followed by a 60 second cleaning cycle.

Results

The combined chemical sensor responses to the six samples (which were determined in replicate) were compared using a quantitative data reduction technique. The results are shown in Figure 9.

Discussion

The embodiment of the chemical sensing system according to the invention demonstrated a clear correlation between the combined chemical sensor responses and the concentration of DIPN in the paperboard. The concentration of DIPN was independently verified using a traditional analytical technique. The dynamic range of the embodiment of the chemical sensing system according to the invention with respect to DIPN is wide, ranging from approximately 3mg/kg to in excess of 70 mg/kg. If an allowable limit for DIPN is to be set, it is expected to be in the range of 4 mg/kg. The repeatability of the measurements obtained was

approximately 5%, which is comparable to other methods which have been used to determine DIPN.

CLAIMS

1. A chemical sensing system for analysing the headspace of a sample comprising a modular sampling unit operatively connected to a chemical sensor assembly,

wherein:

(1) said modular sampling unit comprises:

a mount having an inlet channel and an outlet channel for a carrier gas;

a base member having a hollow interior bound by one or more internal side walls and a basal wall, wherein at least a part of the juncture of the basal wall and one or more internal walls is provided with a supporting collar for supporting the whole or part of the edge of the lower face of the sample;

a closure member having one or more exterior side walls and a basal wall defining a body portion, said body portion being complimentarily shaped with the hollow interior of the base member, wherein the outer edge of the basal wall is provided with an upstanding portion capable of engaging the edge of the upper face of the sample,

wherein in use the base member is inserted into the mount and the body portion of the closure member engages the hollow interior of the base member thereby defining a headspace below the lower face of the sample and a headspace above the upper face of the sample such that the inlet channel communicates with the headspace below the lower face of the sample and the outlet channel communicates with the headspace above the upper face of the sample thereby defining a continuous flow path between the inlet and outlet channel across the majority of the surface area of the sample; and

(2) said chemical sensor assembly comprises:

one or more chemical sensors, each chemical sensor having a chemical sensing component capable of exhibiting a measurable and characteristic response to a chemical stimulus;

a solid body having an entry end for admitting a carrier gas, an exit end for exhausting a carrier gas and one or more compartments for housing each of the one or more chemical sensors therebetween, said one or more compartments being in consecutive fluid communication so as to define a continuous flow path between the entry end and the exit end of the solid body in which the chemical sensing component of the or each chemical sensor may be exposed to the carrier gas.

2. A system as claimed in claim 1 wherein the inlet channel is connected to the headspace below the lower face of the sample by a basal conduit in the basal wall of the base member.

3. A system as claimed in claim 1 or 2 wherein the outlet channel is connected to the headspace above the upper face of the sample by a side conduit in the side wall of the base member.

4. A system as claimed in claim 3 wherein the headspace above the upper face of the sample and the side conduit are connected in fluid communication by a linear tube disposed radially in the upstanding portion of the closure member.

5. A system as claimed in any preceding claim wherein the headspace below the lower face of the sample is in fluid communication with the headspace above the upper face of the sample via one or more substantially U-shaped tubes.

6. A system as claimed in claim 5 wherein a first arm of the U-shaped tube is disposed radially in the supporting collar of the base member and a second arm of the U-shaped tube is disposed radially in the upstanding portion of the closure member, the first and second arm being connected in fluid communication by a connecting portion in the side wall of the base member.

7. A system as claimed in any preceding claim wherein the basal wall of the base member comprises a protrusion for supporting the lower face of the sample.

8. A system as claimed in claim 7 wherein the protrusion converges to a reduced contact end.

9. A system as claimed in any preceding claim wherein the body portion of the closure member is threadedly engaged within the hollow interior of the base member.

10. A system as claimed in any preceding claim wherein the base member is a tight push fit into the mount.

11. A system as claimed in any preceding claim wherein the exterior face of the basal wall of the base member and the mount comprise a locating arrangement to enable correct positioning and insertion of the base member in the mount.

12. A system as claimed in any preceding claim wherein each compartment of the solid body is arranged such that the chemical sensing component is exposed to the carrier gas in a wall jet fashion.

13. A system as claimed in any preceding claim wherein the entry end for admitting a carrier gas and the exit end for exhausting a carrier gas are connected by a substantially linear conduit.

14. A system as claimed in claim 13 wherein the substantially linear conduit defines a spine connecting each of the compartments consecutively.

15. A system as claimed in any preceding claim wherein each compartment is adapted to house the chemical sensing component in substantially free space.

16. A system as claimed in any preceding claim wherein the chemical sensor assembly comprises a plurality of chemical sensors.

17. A system as claimed in any preceding claim wherein the or each chemical sensor is a quartz crystal chemical sensor.

18. A chemical sensor assembly as defined in any of claims 1 to 17.

19. A modular sampling unit as defined in any of claims 1 to 17.

20. A method for detecting the presence of one or more chemical stimuli in the headspace of food packaging material using a chemical sensing system as defined in any of claims 1 to 17, said method comprising:

inserting the base member into the mount;

positioning a sample of the food packaging material in the hollow interior of the base member such that the sample is supported on the supporting collar;

engaging the body portion of the closure member with the hollow interior of the base member;

sweeping the headspace of the sample with a carrier gas;

passing the carrier gas containing one or more chemical stimuli from the headspace to the entry end of the chemical sensor assembly;

measuring the response of the chemical sensors to the carrier gas containing the one or more chemical stimuli from the headspace; and

relating the response to the presence of the one or more chemical stimuli.

21. A method as claimed in claim 20 wherein the chemical stimulus of interest is a DIPN.

22. A method as claimed in claim 20 wherein the chemical stimulus of interest is hexanal.

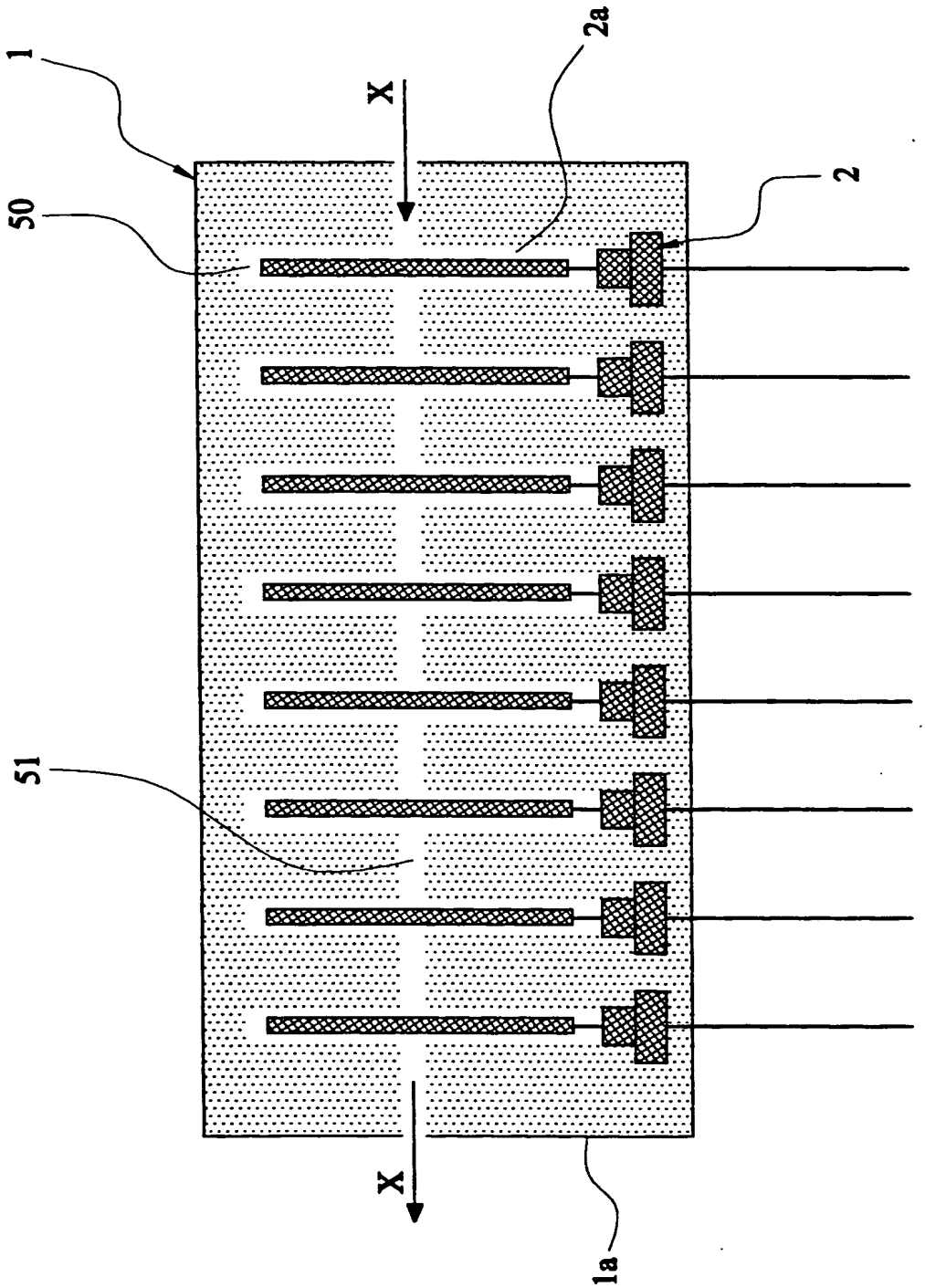

FIG. 1

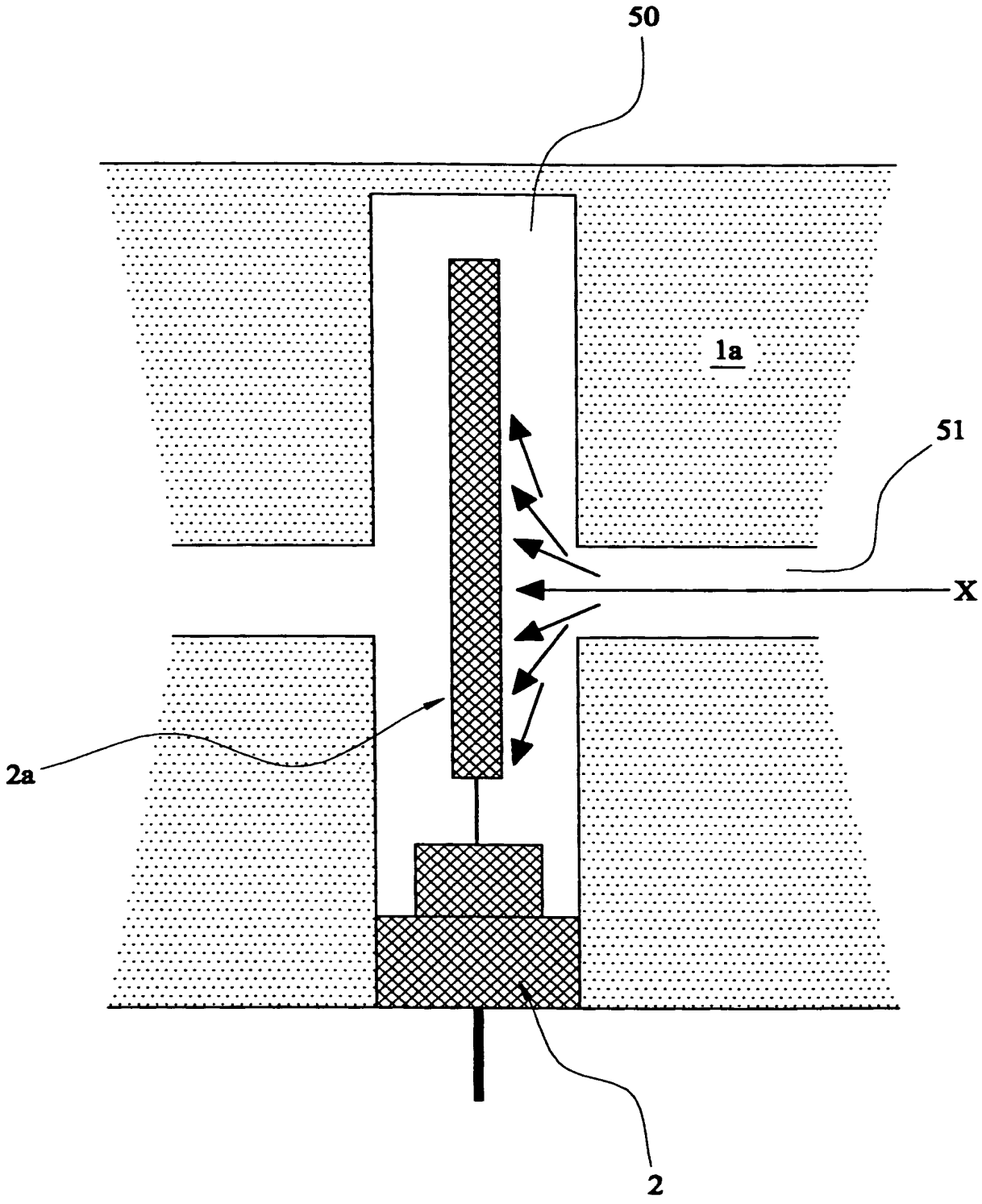

FIG. 2

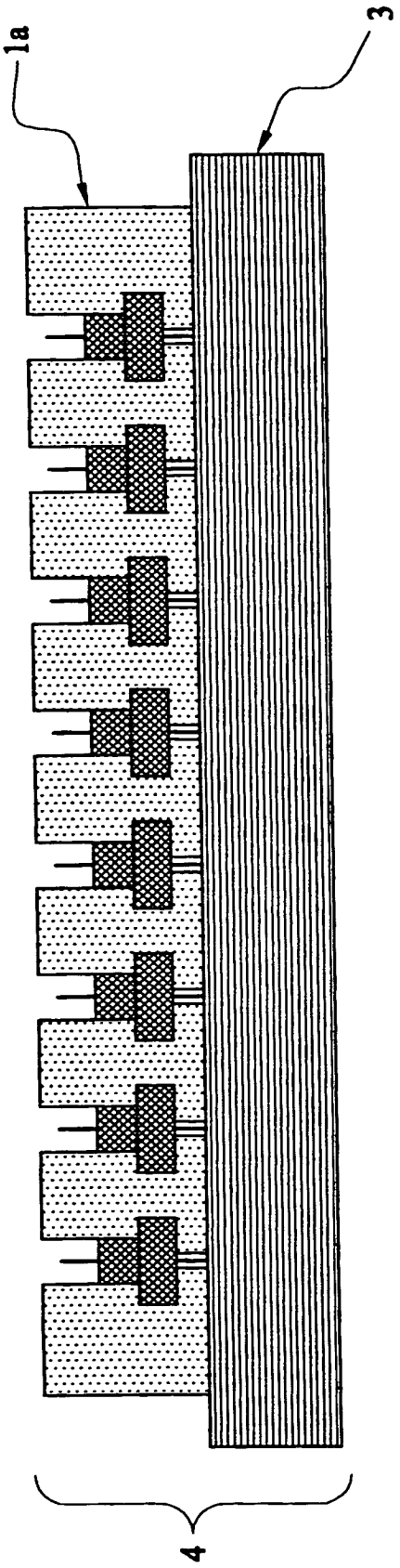

FIG. 3

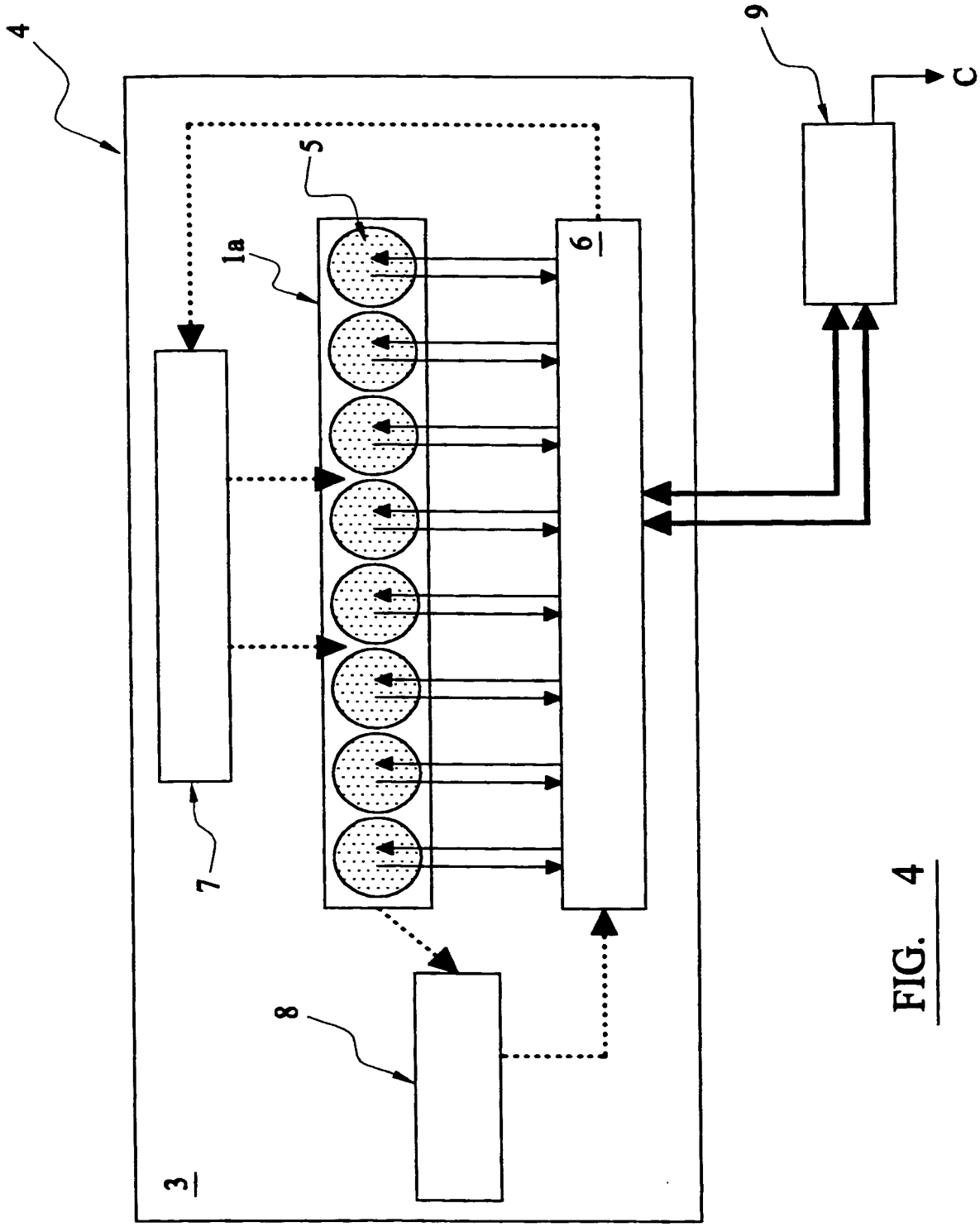

FIG. 4

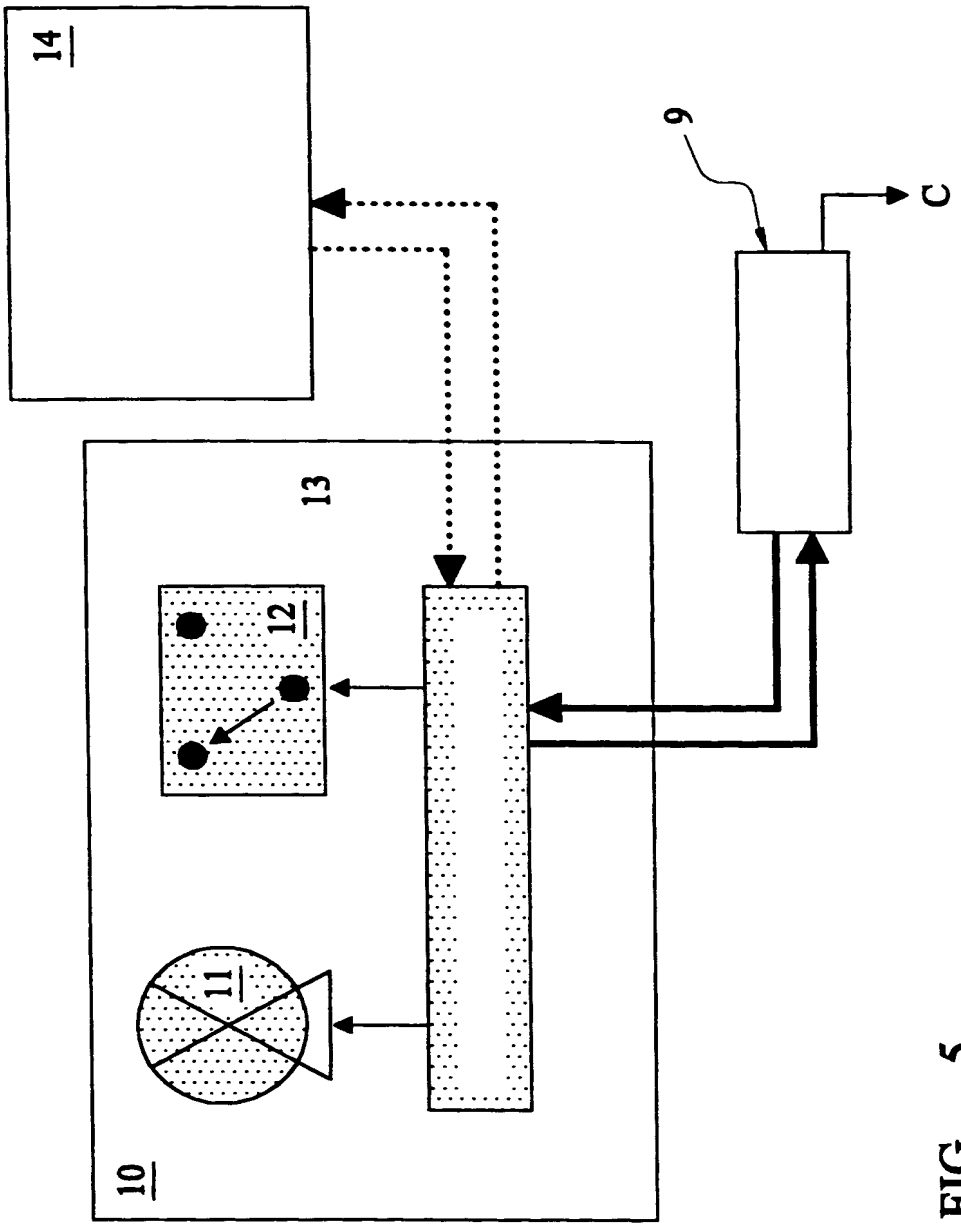

FIG. 5

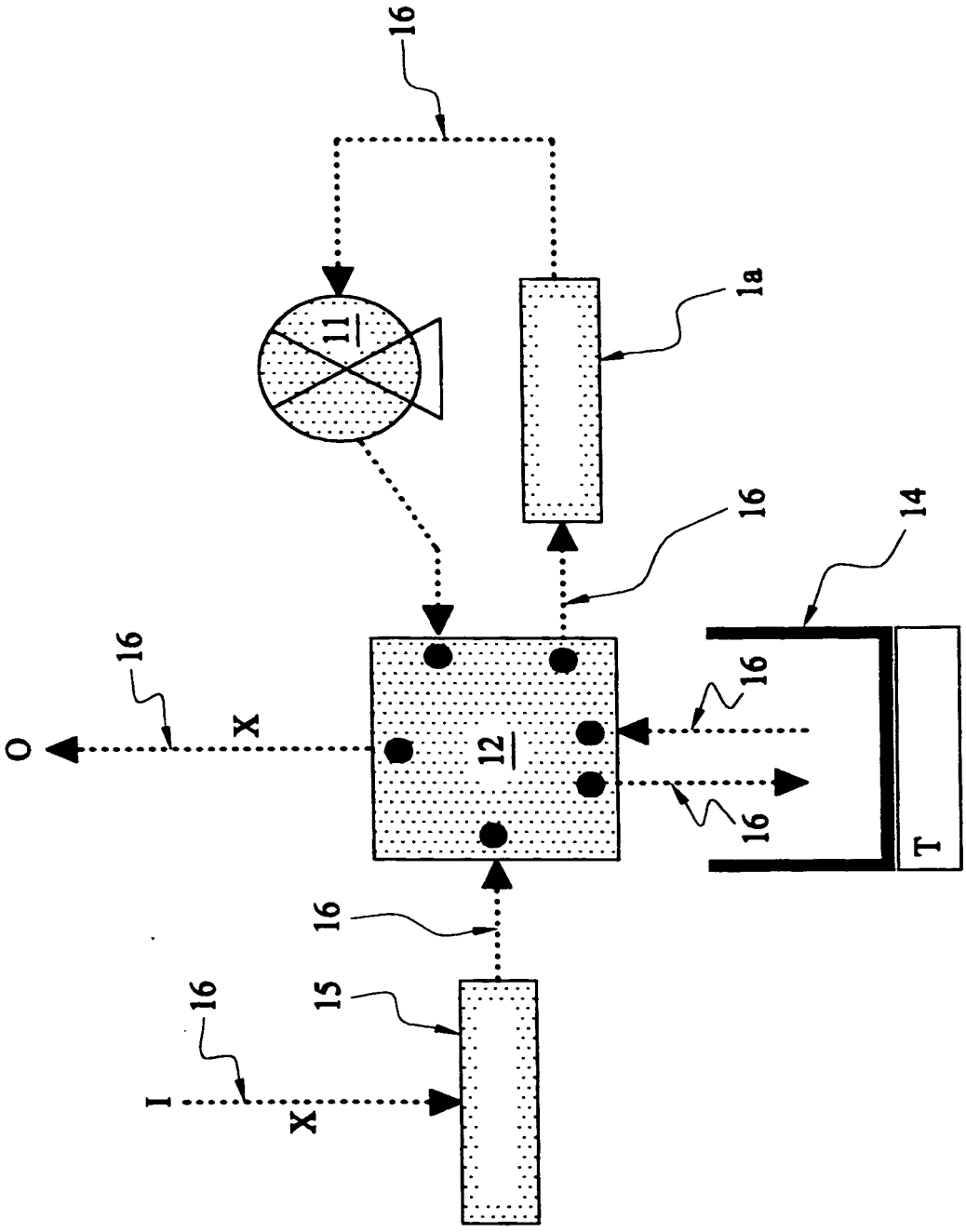

FIG. 6

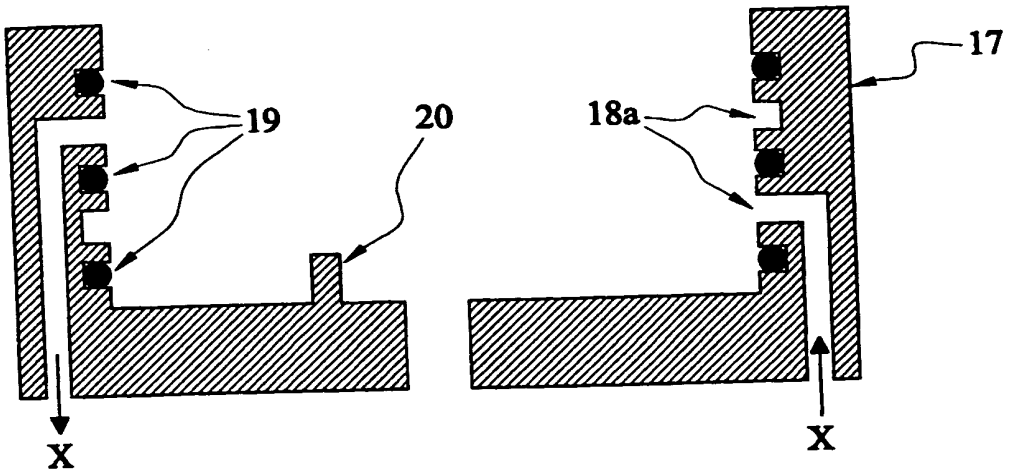

FIG. 7a

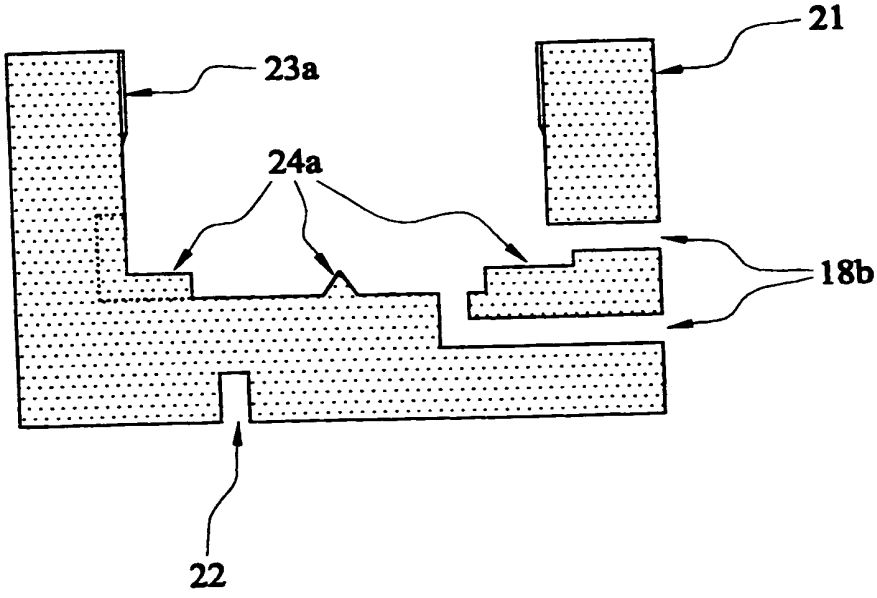

FIG. 7b

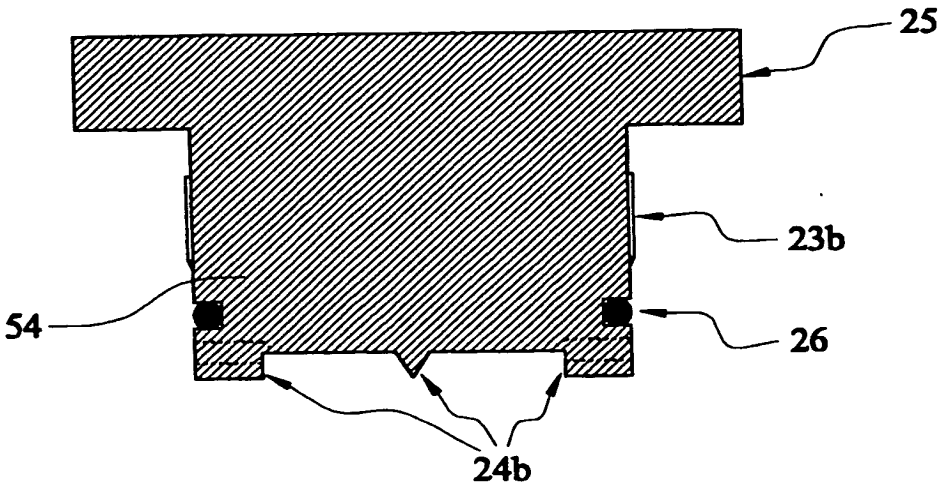

FIG. 7c

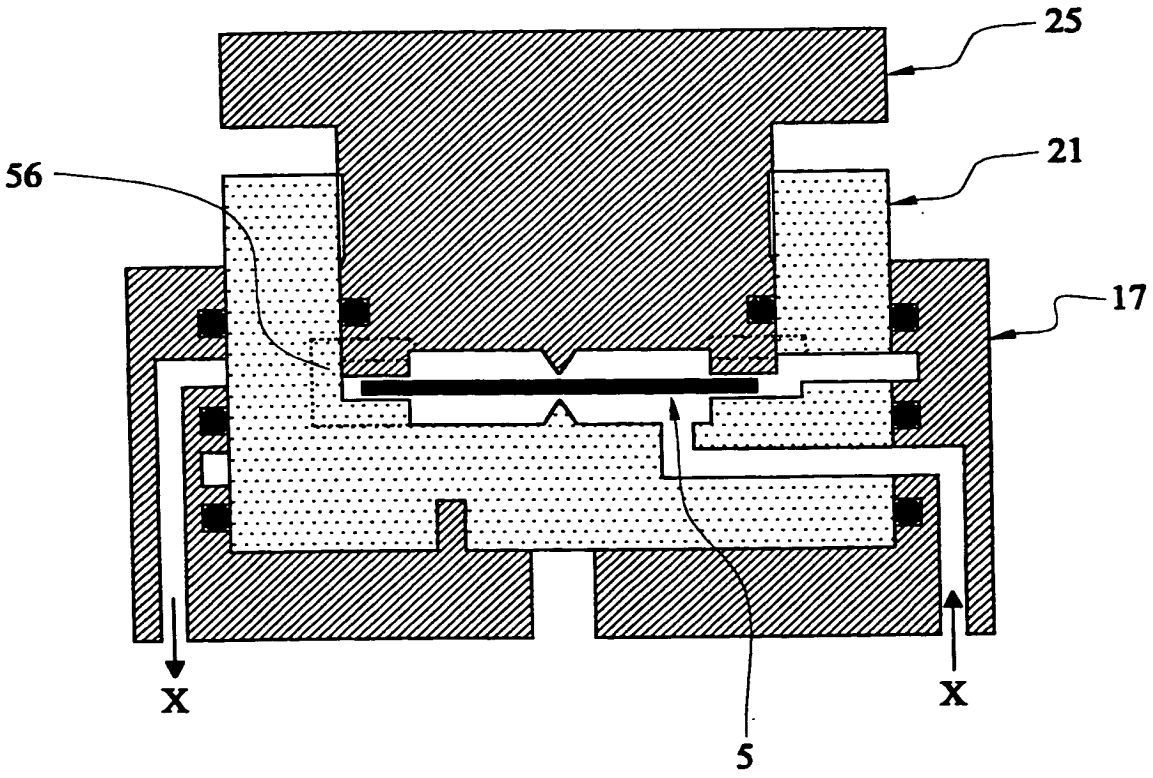

FIG. 7d

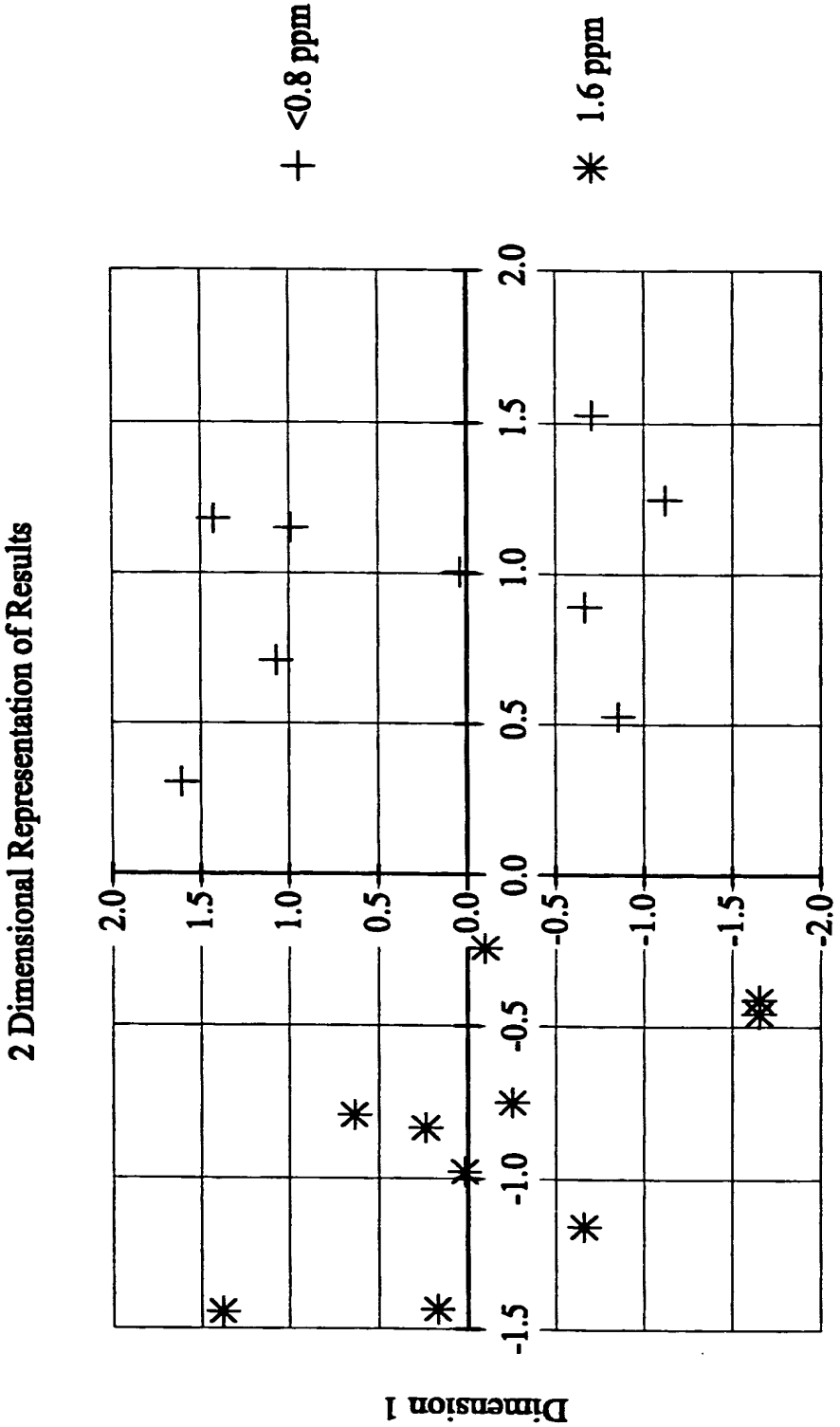
2 Dimensional Representation of Results
<0.8 ppm
1.6 ppm
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
-2.0
Dimension 1
Dimension 2

FIG. 8

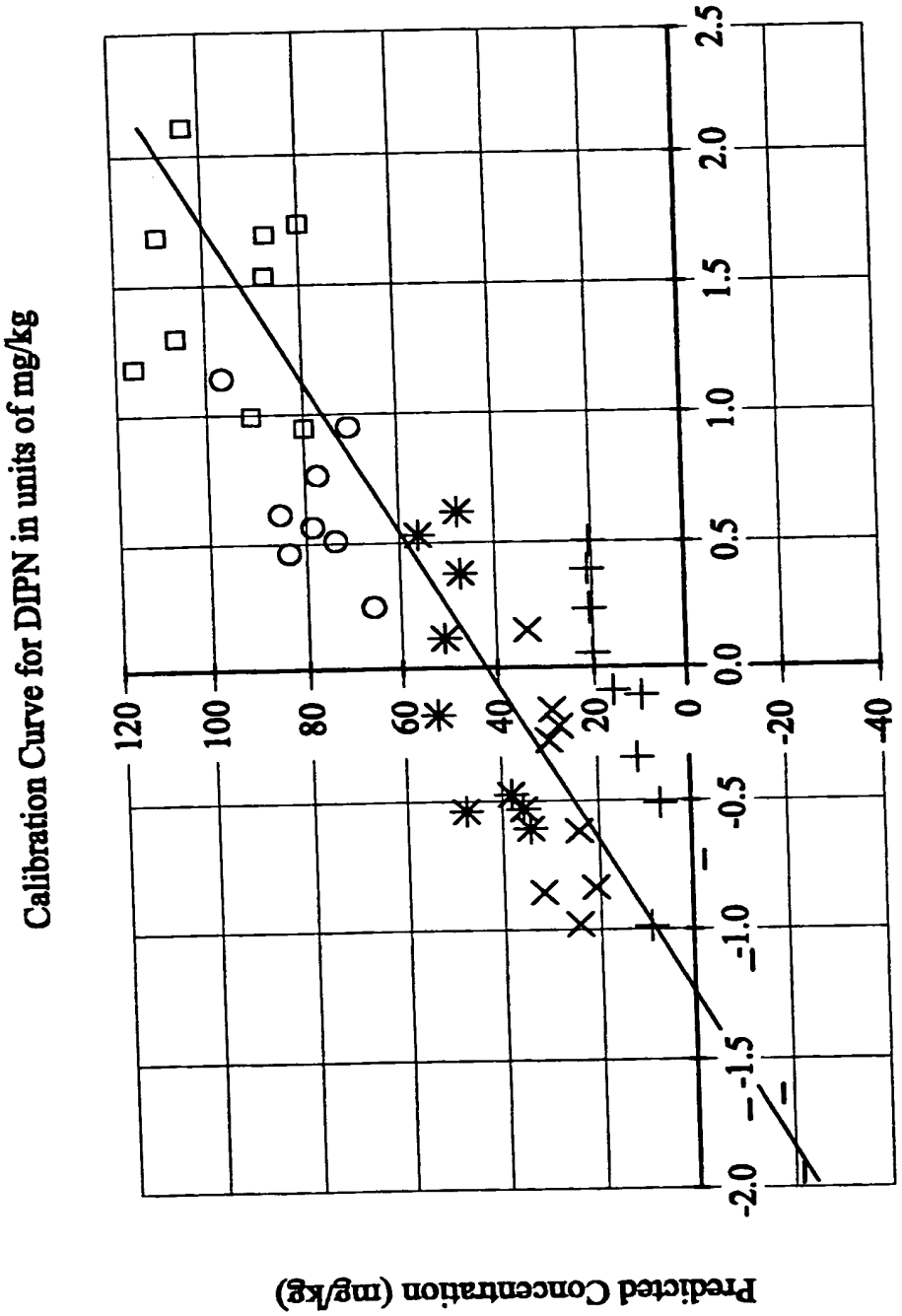
Calibration Curve for DIPN in units of mg/kg
Predicted Concentration (mg/kg)
Dimension 1
120
100
80
60
40
20
0
-20
-40
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
-2.0

FIG. 9

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 00/03664

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 G01N33/00 G01N33/34 G01N1/00 G01N33/02 G01N33/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 567 782 A (HEWLETT PACKARD CO) 3 November 1993 (1993-11-03) column 1, line 8 -column 2, line 29; figure 1 column 3, line 25 -column 4, line 10 column 6, line 41 -column 7, line 32 ---	1,20
A	US 5 482 524 A (NAKANO KAZUO ET AL) 9 January 1996 (1996-01-09) column 2, line 14 - line 35 column 5, line 11 - line 57; figure 1 ---	1,20
A	US 5 469 369 A (ROSE-PEHRSSON SUSAN L ET AL) 21 November 1995 (1995-11-21) column 3, line 48 - line 60 column 4, line 37 - line 66 column 8, line 35 - line 50 column 11, line 59 - line 65 ---	17
	-/--	

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
1 March 2001	07/03/2001

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Joyce, D

3

Form PCT/ISA/210 (second sheet) (July 1992)

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 2 184 037 A (FURNITURE IND RES ASS) 17 June 1987 (1987-06-17) page 1, column 1, line 7 -page 1, column 2, line 122	1,20
A	OGURI N ET AL: "DEVELOPMENT OF A CURIE-POINT HEADSPACE SAMPLER FOR CAPILLARY GAS CHROMATOGRAPHY" JOURNAL OF HIGH RESOLUTION CHROMATOGRAPHY,WILEY VCH, WEINHEIM,DE, vol. 14, no. 2, February 1991 (1991-02), pages 79-82, XP000200269 ISSN: 0935-6304 the whole document	1
A	WO 97 46755 A (AXRUP LARS ;NILSSON TORSTEN (SE); KORSNAES AB PUBL (SE)) 11 December 1997 (1997-12-11) page 1, line 26 -page 3, line 19	22

INTERNATIONAL SEARCH REPORT

Information on patent family members

nati[redacted]pplication No
PCT/GB 00/03664

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0567782 A	03-11-1993	US 5363707 A	15-11-1994
		JP 6043075 A	18-02-1994
US 5482524 A	09-01-1996	JP 7083808 A	31-03-1995
US 5469369 A	21-11-1995	NONE	
GB 2184037 A	17-06-1987	NONE	
WO 9746755 A	11-12-1997	SE 504875 C	20-05-1997
		AU 3113597 A	05-01-1998
		CA 2257215 A	11-12-1997
		EP 0907789 A	14-04-1999
		SE 9602225 A	20-05-1997

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

STUTTARD, Garry Philip
Urquhart-Dykes & Lord
Tower House
Merrion Way
Leeds LS2 8PA
GRANDE BRETAGNE

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT
(PCT Rule 71.1)

Date of mailing *(day/month/year)*	29.10.2001
Applicant's or agent's file reference GS/AMW/P207043WO	**IMPORTANT NOTIFICATION**

International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/GB00/03664	25/09/2000	25/09/1999

Applicant
QUALITY SENSOR SYSTEMS LTD. et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. **REMINDER**

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/	Authorized officer
European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Götz, K Tel.+49 89 2399-7381

Form PCT/IPEA/416 (July 1992)

PATENT COOPERATION TREATY

PCT

REC'D 31 OCT 2001
WIPO PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

14

Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
GS/AMW/P207043WO		
International application No.	International filing date *(day/month/year)*	Priority date *(day/month/year)*
PCT/GB00/03664	25/09/2000	25/09/1999
International Patent Classification (IPC) or national classification and IPC G01N33/00		
Applicant QUALITY SENSOR SYSTEMS LTD. et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 8 sheets, including this cover sheet.

   ☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

   These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

   - I ☒ Basis of the report
   - II ☐ Priority
   - III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   - IV ☒ Lack of unity of invention
   - V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   - VI ☐ Certain documents cited
   - VII ☒ Certain defects in the international application
   - VIII ☒ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
12/04/2001	29.10.2001
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Diez Schlereth, D Telephone No. +49 89 2399 7488

Form PCT/IPEA/409 (cover sheet) (January 1994)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/03664

I. Basis f the r port

1. With regard to the el ments of the international application *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17))*:
Description, pages:

1-22 as originally filed

Claims, No.:

1-22 as originally filed

Drawings, sheets:

1/10-10/10 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

IV. Lack of unity of invention

1. In response to the invitation to restrict or pay additional fees the applicant has:

☐ restricted the claims.

☐ paid additional fees.

☐ paid additional fees under protest.

☐ neither restricted nor paid additional fees.

2. ☐ This Authority found that the requirement of unity of invention is not complied and chose, according to Rule 68.1, not to invite the applicant to restrict or pay additional fees.

3. This Authority considers that the requirement of unity of invention in accordance with Rules 13.1, 13.2 and 13.3 is

☐ complied with.

☒ not complied with for the following reasons:
see separate sheet

4. Consequently, the following parts of the international application were the subject of international preliminary examination in establishing this report:

☒ all parts.

☐ the parts relating to claims Nos. .

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-22
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-17,19-22
	No:	Claims	

Industrial applicability (IA)	Yes:	Claims	18
	No:	Claims	

2. Citations and explanations
see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:
see separate sheet

VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:
see separate sheet

Re It m IV

The requisite for unity of invention (Rule 13.1 PCT) is not fulfilled, since a novel and inventive technical relationship involving one or more of the same of corresponding special technical features in the sense of Rule 13.2 PCT does not exist between the subject-matter of independent claim 18 and independent claims 1 and 19-20 (see item V. below). The separate inventions are:

claims 1-17 and 19-22: modular sample unit, chemical sensing system, and method for detecting chemical stimuli in the headspace of packing material.

claim 18: chemical sensor assembly.

However, in order to accelerate the procedure and since a complete Search Report has been issued by the International Search Authority, a written opinion is given for all groups of inventions.

Re Item V

1.) Reference is made to the following documents:

D1: WO-A-97/46755
D2: US-A-5,482,524
D3: GB-A-2 184 037
D4: EP-A-0 567 782
D5: N. Oguri et al (1991) HRC J. High Resol. Chromat., 79-82
D6: US-A-5,469,369

2.) The subject-matter of claims 1-17 and 19-22 is considered to be novel and inventive within the sense of Art. 33 (2) and (3) PCT, for the following reasons:

D1, which is considered to be the closest prior art as regards claim 1, discloses a chromatographic system (and a method) for detecting volatile organic compounds "VOCs" (e.g. hexanal) emitted by finished paper products. Paper samples are subjected to a thermal desorption treatment in a chamber, desorbed VOCs are carried

with a carrier gas to an adsortion column, from which they are thermally desorbed and transported in a carrier gas stream to the chromatographic unit (p. 1, l. 5-10; p. 2, l. 10-32; p. 3, l. 1-23; p. 4-6; p. 11, l. 5-28).

D2 discloses a system (and a method) for detecting impurities in the surface of a solid planar substrate (e.g. a silicon wafer), in which the sample is placed in a desorption chamber and subjected to a thermal desorption process. The impurities, which are delivered to the headspace of the chamber defined above the substrate (provided with an inlet and an outlet for flushing carrier gas into said headspace) are transported by a carrier gas stream to a measuring unit (col. 2, l. 18-24; Embodiment 1; Fig. 1).

D3 discloses a system (and a method) for measuring the rate of formaldehyde emission from a resin-bonded or -impregnated material (e.g. paper), in which the sample is introduced within a closed container having an air inlet and outlet to transport the emitted formaldehyde to a series of absorbing filters (p. 1, l. 5-15; p. 3, l. 64-98; p. 4, l. 10-67; Fig. 1).

D4 discloses a headspace sample unit, in which a sealed sample container is inserted into the unit, such as to communicate the sample headspace of the container with an external headspace of the unit, said external headspace having a gas inlet and outlet to transport the material to a chromatograph (col. 2, l. 45-58; col. 3, l. 1-15; col. 4, l. 11-31; col. 5, l. 1-27; Fig. 1). D5 discloses a Cuire-Point headspace sampler for capillary gas chromatography (Figs. 1-2).

D6 discloses a system for detection of traces of volatile organic compounds based on the use of a surface acoustic wave sensor array. Gaseous sample is directed to the sensor array through a manifold that includes at least one preconcentrator tube which basically consists on an adsorbent column from which the sample is recovered by thermal desorption (col. 4, l. 48-67; col. 5, l. 1-37; Figs. 1-2).

The systems disclosed in D1-D3 and D6 do not have a sampling unit in which the sample is inserted such as to define a headspace above and below the sample. D4-D5 disclose sampling units in which the gas inlet and outlet are located in the same headspace. In all the systems disclosed in D1-D6 the gas carrier flow path does not go through the sample, but flows over/below the sample.

A sampling unit as defined in independent claim 1 defines a headspace above the upper face of the sample and a headspace below the lower face of the sample, wherein the inlet channel for carrier gas communicates with the lower headspace and the outlet channel for carrier gas communicates with the upper headspace.

This configuration of the sampling unit establishes unavoidably a continuous flow path for the carrier gas which crosses through the majority of the surface area of the sample, while minimizing the sampling dead volume operatively connected to the sensor array. The sensing system of claim 1 provides fast on-line measurements at high sensitivity.

The skilled person equipped with the teaching of D1-D6 would have had neither motivation nor technical guidance to modify the system of D1 arriving at a detection system as claimed in claim 1 (and 2-17 as dependent thereon).

The subject-matter of claims 19 and 20-22 is considered to be novel and inventive within the sense of Art. 33 (2) and (3) PCT, for analogous reasons as discussed above.

3.) In the light of the known prior art (D1-D6), the subject-matter of claim 18 is novel (Art. 33 (2) PCT), but is not considered to be inventive within the sense of Art. 33 (3) PCT, for the following reasons:

D6, which is considered to be the closest prior art as regards claim 18, discloses a chemical sensing assembly in which an array of surface acoustic wave sensor devices are housed inside a chamber (solid body having one compartment) having a gas inlet and outlet (see Fig. 1). The subject-matter of claim 18 differs therefrom in that inside the housing (solid body) each of the sensors of the array is housed within a compartment, wherein each compartment has a gas inlet and outlet, such that the compartments are in successive fluid communication defining a continuous flow path between the entry and the exit of the housing (solid body).

Since the configuration of the sensor array of D6 also provides a continuous flow path between the entry and the exit of the housing containing the array, it is considered that a compartimentalization of the housing as defined in claim 18 is a constructional alternative to the device of D6, which comes within the scope of the customary practice followed by persons skilled in the art, which does not seem to result in any unexpected

technical effect.

Re Item VII

Contrary to the requirements of Rule 5.1 (a) (ii) PCT, the relevant background art disclosed in the documents D1-D6 is not mentioned in the description, nor are these documents identified therein.

Re Item VIII

For the sake of clarity (Art. 6 PCT), the abbreviation DIPN used in claim 21 should have been written in its full form (see page 11).

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference	**FOR FURTHER ACTION** see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
GS/P207043WO		
International application No.	International filing date *(day/month/year)*	(Earliest) Priority Date *(day/month/year)*
PCT/GB 00/ 03664	25/09/2000	25/09/1999
Applicant		
QUALITY SENSOR SYSTEMS LTD. et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

[X] It is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the report**

   a. With regard to the **language,** the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

   [ ] the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

   b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

   [ ] contained in the international application in written form.

   [ ] filed together with the international application in computer readable form.

   [ ] furnished subsequently to this Authority in written form.

   [ ] furnished subsequently to this Authority in computer readble form.

   [ ] the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

   [ ] the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. [ ] **Certain claims were found unsearchable** (See Box I).

3. [ ] **Unity of invention is lacking** (see Box II).

4. With regard to the **title,**

   [X] the text is approved as submitted by the applicant.

   [ ] the text has been established by this Authority to read as follows:

5. With regard to the **abstract,**

   [X] the text is approved as submitted by the applicant.

   [ ] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. 6

   [X] as suggested by the applicant.

   [ ] because the applicant failed to suggest a figure.

   [ ] because this figure better characterizes the invention.

   [ ] None of the figures.

INTERNATIONAL SEARCH REPORT

International Application No
P[redacted]GB 00/03664

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 G01N33/00 G01N33/34 G01N1/00 G01N33/02 G01N33/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 567 782 A (HEWLETT PACKARD CO) 3 November 1993 (1993-11-03) column 1, line 8 -column 2, line 29; figure 1 column 3, line 25 -column 4, line 10 column 6, line 41 -column 7, line 32 ---	1,20
A	US 5 482 524 A (NAKANO KAZUO ET AL) 9 January 1996 (1996-01-09) column 2, line 14 - line 35 column 5, line 11 - line 57; figure 1 ---	1,20
A	US 5 469 369 A (ROSE-PEHRSSON SUSAN L ET AL) 21 November 1995 (1995-11-21) column 3, line 48 - line 60 column 4, line 37 - line 66 column 8, line 35 - line 50 column 11, line 59 - line 65 ---	17
	-/--	

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
1 March 2001	07/03/2001

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Joyce, D

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INTERNATIONAL SEARCH REPORT

International Application No
[redacted]GB 00/03664

C.(Continuation) DOCUMENTS CONSIDER[redacted]TO BE RELEVANT

Category °	Citation of document, with indication,where appropriate, of the relevant passages	Relevant to claim No.
A	GB 2 184 037 A (FURNITURE IND RES ASS) 17 June 1987 (1987-06-17) page 1, column 1, line 7 -page 1, column 2, line 122 ---	1,20
A	OGURI N ET AL: "DEVELOPMENT OF A CURIE-POINT HEADSPACE SAMPLER FOR CAPILLARY GAS CHROMATOGRAPHY" JOURNAL OF HIGH RESOLUTION CHROMATOGRAPHY,WILEY VCH, WEINHEIM,DE, vol. 14, no. 2, February 1991 (1991-02), pages 79-82, XP000200269 ISSN: 0935-6304 the whole document ---	1
A	WO 97 46755 A (AXRUP LARS ;NILSSON TORSTEN (SE); KORSNAES AB PUBL (SE)) 11 December 1997 (1997-12-11) page 1, line 26 -page 3, line 19 -----	22

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Form PCT/ISA/210 (continuation of second sheet) (July 1992)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
P[redacted]GB 00/03664

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0567782 A	03-11-1993	US 5363707 A	15-11-1994
		JP 6043075 A	18-02-1994
US 5482524 A	09-01-1996	JP 7083808 A	31-03-1995
US 5469369 A	21-11-1995	NONE	
GB 2184037 A	17-06-1987	NONE	
WO 9746755 A	11-12-1997	SE 504875 C	20-05-1997
		AU 3113597 A	05-01-1998
		CA 2257215 A	11-12-1997
		EP 0907789 A	14-04-1999
		SE 9602225 A	20-05-1997